

Insulating Biomaterials N01-NS-2-2347

Tenth Quarterly Progress Report Jan-March, 2005

National Institutes of Health

National Institute of Neurological Disorders and Stroke

Neural Prosthesis Program



InnerSea Technology

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The goal of the Insulating Biomaterials work is to identify and evaluate materials, coatings, and assembly techniques suitable for protection of integrated circuit devices being considered for neural prosthetic applications.

Instrumentation Systems

Accelerated detection of degradation is the main tool for studying materials for implantable devices. The new Passivation Test System consists of 4 major components: the Tube Top, the Measurement Unit, the Data collection Unit, and the Calibration Unit. These components are described below. Basically, as illustrated in Figure 1 the device to be tested is placed into the saline soak tube.

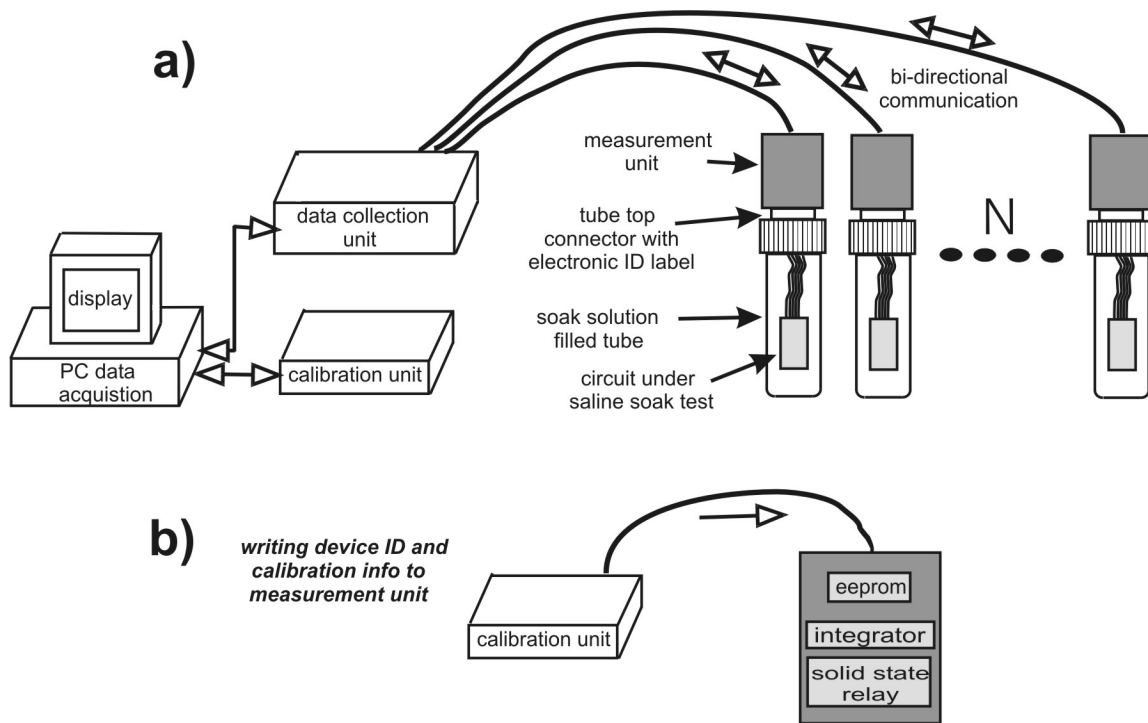


Figure 1: Cartoon showing basic elements of new test system.

1) the Tube Top - This provides: a physical attachment point for the device under test; the electrical connections to the system; and an EEPROM that contains information about the particular device in that is under soak test in that respective tube.

2) the Measurement Unit - This contains: the analog circuitry required to perform the measurement; a calibration check unit; a continuity tester for triple



track devices; and another EEPROM which contains required calibration information.

3) the Data Collection Unit - This accepts the data (consisting of timing signals) from the measurement units, converts them into leakage values, and transmits those values to a host computer.

4) the Calibration Unit - This is a standalone unit that is used with each device before any testing at all is performed, and it is normally used only at the onset of testing. Thus, if a device is tested each day for 100 days, the Calibration Unit is “hooked” up to Measurement Unit at the start of day 1 so that device identification data can be downloaded to the EEPROM that is located on the Tube Top, along with the calibration information particular to the device under test and that will be needed for the Measurement Unit to interpret the measured parameters for every succeeding test day.

Recent Results:

During this period we concentrated on creating the data collection software for the system. The system uses the TCP/IP to communicate with the host computer, so our applications need to be able to accept incoming connections, recognize the source of the data, and parse the data stream for recording in the database. The system is device centric – meaning that the system doesn’t care which Data Collection unit (DCU) the Device Under Test (DUT) is connected to, instead only that the device is present somewhere in the measurement chain. When a jar is connected to the system, a record of the device’s identifier is added to the database, and subsequent data is indexed under that identifier. This will eliminate problems we have seen with the current system which is location centric – meaning that the data is indexed by location in the system. This method is operator intensive since the operator is required to maintain the device location table, and therefore is prone to errors. The new system will keep track of a device’s location only to aid in physically finding the DUT, and to log the current testing temperature. The program is currently being developed using Agilent Vee Pro Version 7. Vee allows for quicker development time, and allows the use of



COM and .NET objects in the program. COM and .NET objects are external libraries that expand the available functionality of Vee, and allow much greater flexibility in designing a program. In this case we use the Microsoft Windows Sockets control for TCP/IP connectivity, and Microsoft ADODB database connectivity control for control over our database. In addition we reuse functions originally written for the current passivation test system.

To meet these ends, some changes to the firmware were necessary. The module which accepted and transmitted data to the xPort (Used to provide Ethernet connectivity) was rewritten in assembly language to improve performance. The function to stop a currently running test was also modified, because it was only working intermittently.

Peel Testing

A variety of tests were completed and/or recently summarized following soak times of up to 2 years. The following preliminary reports were from the developmental work associated with the test itself, and will be repeated in more detail to study the materials of interest. All peel testing is currently being done using Nusil silicones.

FGQuartz

3/11/02

The purpose of this test was to determine if there was a difference in adhesion between glass and quartz slides.

Two glass and 2 quartz slides were cleaned along with 4 aluminum slides. The glass and quartz slides were adhered to the aluminum slides using MED4-4220 and baked. Lengths of .003 fiberglass tape were adhered to the slides and baked.

The results for the 4 samples are:

Substrate	Average
Glass 1	438.18 gms
Glass 2	2637.88 gms



Quartz 1	2625.76 gms
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Quartz 2	2446.12 gms
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Except for Glass 1, which stopped pulling from the silicone and started pulling from the glass after a year, the other three samples were fairly equal and no advantage was shown for either substrate.

LCP – Auburn	10/15/03
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Four glass slides were cleaned and adhered to clean aluminum slides using MED4-4220 and baked. Four samples of processed LCP were attached to the slides using MED4-4220 and baked. Strips of kapton tape were laid perpendicularly to the slides and lengths of .007 fiberglass tape were adhered to the striped slides using MED4-4220 and baked.

The pull force results for the four samples are:

LCP Sample	Average Pull Force
Shiny Ion	1513.59 gms
Matte Ion	1130.69 gms
Shiny Matrix	736.66 gms
Matte Matrix	579.43 gms

Shiny and Matte describe the surface of the LCP – Shiny is virgin LCP while Matte has been micro-roughened to adhere copper which was subsequently etched off before use in this study. Ion is an ion cleaning procedure using Argon bombardment. Matrix refers to a solvent sequence of cleans.

The Ion Clean and Shiny samples showed the greater adhesion results overall.

LCP Pull Test / unprocessed LCP July 12, 2004

Two pull test samples were prepared using the usual base of aluminum slides adhered to glass slides with MED4-4220. Two lengths of .003 fiberglass tape were secured to a teflon pad with more MED4-4220 and samples of unprocessed LCP approximately equal to the width of the tape were laid on the tape. The



glass slide was covered with MED4-4220 and laid on top of the tape/LCP section and pressed into place. The entire unit was then covered with MED4-4220 and baked to cure.

The pull testing lasted approximately 2 years and the samples differed slightly in average pull force, but not significantly. Sample #1 averaged 146 grams pull force and sample two averaged 106. Neither sample, then, created a strong bond with the silicone.

MED4750/MED4-4220

Final Report

2/16/04

This study was undertaken to determine the adhesion of a slab of MED4750 with MED4-4220. The purpose is to identify appropriate conditions for sealing silicone coated lead wires into a silicone encapsulation.

Sample was created by cutting a slab of MED4750 the size of aluminum slide (1"x3") and cleaning slab and slide. The two were adhered using MED4-4220 and baking. Kapton strips were applied and MED4-4220 spread on the sample and .007 fiberglass tape adhered. Sample was then baked.

Results show an average pull force over all tests to be 1505.83 grams.

MED4750 – CSM4220-3

Final Report

2/18/04

This study was undertaken to determine the adhesion of CSM4220-3 to a slab of MED4750. The purpose is to identify appropriate conditions for sealing silicone coated lead wires into a silicone encapsulation. The slab of MED4750 was adhered to an aluminum slide and then .007 fiberglass tape was adhered to the slab using CSM4220-3.

The results were typical of CSM4220-3 – roughly 250-300gm pull force on average. The tests were halted as no variation was seen in this sample as with most CSM4220-3 studies.

MED4-4220 Mix – No Mix #2

Final Report

4/10/02



This study was undertaken to determine whether the mixing of the silicone affects adhesion.

Four samples were created with microscope slides adhered to aluminum slides and lengths of .003 fiberglass tape adhered to these units with MED4-4220. One of the samples applied the silicone to the slide without mixing – directly from the sxs kit. The other three were adhered after the silicone was mixed and applied to the slide with a glass rod.

Though the sample that was not mixed, was terminated early due to the tape ripping, it had significantly greater pull force than the mixed samples. The mixed samples had average pull forces of around 500 gm over the entire study of almost three years. The sample that was not mixed was at 2000 gm when it was terminated.

Tape Test – Large Weave

9/11/04

This test was initiated to determine whether a larger weave tape would produce better/more consistent adhesion results.

Glass and aluminum slides were cleaned and attached with room temp. MED4-4220 and baked. After cooling, Kapton strips were placed perpendicularly on the glass slide at 1cm intervals. Lengths of large weave tape were then adhered to the glass slide with the strips and baked. Four samples were prepared.

The results are fairly consistent between the samples:

LW1 = 1890.94 gms/average

LW2 = 1944.76 “

LW3 = 2125.36 “

LW4 = 2009.65 “

1992.68 gms. average all samples

CSM4220-3 Vacuum

2/28/04



This was an adhesion study to determine whether placing the silicone (CSM4220-3) in a vacuum chamber would effect the adhesion.

A glass microscope slide and aluminum slide were cleaned. A length of fiberglass tape (.003") was cut and spread with CSM4220-3 adhering it to a Teflon pad. More silicone was then spread on top of the tape. This unit was then placed in a vacuum chamber for 10 minutes. The unit was then completed normally.

Results show no significant improvement in adhesion. The average pull force for all samples was 63.20 gms.

Centrifuge/Quartz

5/16/02

This was an adhesion study to determine whether centrifuging the silicone (MED4-4220) would increase the adhesion.

Two quartz slides were adhered to aluminum slides, after cleaning, using MED4-4220 and then baked. One slide was adhered to fiberglass tape with MED4-4220 directly from the SXS kit. Another amount of MED4-4220 was centrifuged for 3 minutes and applied to the slide using a glass stir rod. The sample was completed as usual and baked.

The average pull force for all tests with the centrifuged MED4-4220 was 2917.27 gms. For the control sample the average pull force for all tests was 2511.18 gms.

It appears that the centrifuging had an affect on the adhesion properties of the MED4-4220, whether from centrifuging or warming. The removal of bubbles from the silicone appears to have been the result of the centrifuging. Warming of the silicone was also a result of the centrifuging and may have an effect on the adhesion.

R2188 Adhesion Test

10/2/02



This test was to determine the adhesion properties of R2188 on glass and quartz slides.

Two glass and 2 quartz slides were cleaned and adhered to clean aluminum slides using R2188 and baked. Lengths of .007 fiberglass tape were adhered to the slides and baked.

The results of the tests were:

Substrate	Average
Glass 1	75.17 gms
Glass 2	48.43 gms
Quartz 1	141.75 gms
Quartz 2	143.3 gms

Though no sample has significant pull force as compared to other silicones, the quartz showed a slight improvement over glass in pull force.

Summary:

From this testing a well defined assembly and test protocol has now been established, and is now being used to study various adhesion promoters on glass, quartz and LCP. Results should begin to be available next quarter. CSM4-4220 and MEDD4-4220 will be studied extensively with various silane based adhesion promoters since these materials have inherently different adhesion properties and handling properties yet are of very similar chemistry.

IDE Testing for Flux Contamination and Adhesion Promotion



In the period of 1/05 through 3/05, four experiments had been conducted on EFITTALUM to reduce flux contamination problems. The experiment procedures and results are summarized as follows.

EFITT050119ALUM

For all the experiments conducted so far, solder flux contamination has been the major cause to failure. One assumption is that solder flux may trapped under the insulation of the wires; therefore, bare wire was proposed to replace Teflon insulated Belden wire. In this experiment, two (Electro Films Incorporated Triple Track) EFITT designs (3 mil lines and spaces, 1cm active area, 4 lead attachment) on alumina (EFITT050119ALUME04&E06) were prepared. 26AWG bare Cu wire (628-026) was solder onto contact pads with Kester water soluble solder. The solder joints were first rinsed with warm tap water and DIH₂O, and blow-dried with N₂. The samples were then cleaned with following procedure:

- Ultra sonicate (US) in DIH₂O for 5min @ 45⁰C. Blow-dry with N₂.

Rinse with Chemtronics Flux-Off Aqueous (CTA). Blow-dry with N₂.

US in CTA1 for 5min @ 45⁰C.

Rinse with CTA. Blow-dry with N₂.

US in CTA2 for 5min @ 45⁰C. Blow-dry with N₂.

Rinse with DIH₂O. Blow-dry with N₂.

US in Vertrel XMS Plus (MS-797) for 5 min. Blow-dry with N₂.

US in IPA for 5min @ 45⁰C. Blow-dry with N₂.

After cleaning, the samples were baked at 150⁰C for 5 min and cooled under the hood before sliding Silastic Lab Tubing (0.058"ID, 0.077"OD) from Dow Corning over the bare wires. The devices were finally encapsulated with CSM4220-3(Lot# 34302), vacuumed to remove trapped air, and filled in the silicone tubing with CSM4220-3 silicone. For EFITT050119ALUME06, the silicone was cured at 150⁰C for 3 hour. For EFITT050119ALUME04, the sample was shifted upward during curing process such that surface of TT was only thinly covered. After it



was baked for half hour at 125⁰C, the sample was taken out, trimmed, over coated with CSM4220-3 one more time, and baked at 150⁰C for 2.5 hour. However, this repairing process may do more damage to the silicone coating than help for silicone may not be fully cured at 125⁰C for half hour and the trimming may tear the silicone from the substrate. As showed in a later microscopic exam, corrosion due to electroplating is found at the bottom edge of TT on EFITT050119ALUME04 while it is corrosion free for TT of EFITT050119ALUME06. . On 1/21/05, EFITT050119ALUME04&E06 were put under dry test. Three-day measurement showed all of the devices had resistance at 10¹² Ω. After PB ringer was added to the test tube on 1/24/05, EFITT050119ALUME04 only passed one cycle of leakage measurement at 10⁹ Ω with full sweeping range (-5, +5V) and EFITT050119ALUME06 lasted two cycles also at 10⁹ Ω with full sweeping range before failing. Pass test results are summarized in Table 6. Microscopic study showed the dendrite corrosion at the solder joints for both devices, apparently due to the residue of solder flux that hindered the adhesion of silicone at the contacts. Corrosion at solder joints also extended onto the leads. TT of EFITT050119ALUME06 was well protected; however, electroplating corroded part of TT on EFITT050119ALUME04 as mentioned earlier.

EFITT050203ALUMCSM4220-3&MED4220, EFITT050204ALUMCSM4220-3&MED4220

Besides trying to control solder flux contamination, silicone adhesion promoter, vinyltriethoxysilane (VTES) coupling agent from Gelest Inc. (Lot# 38-2520) was applied for improving silicone adhesion on EFITTALUM substrates. Two sets of devices were assembled EFITT050203ALUMCSM4220-3 & MED4220 and EFITT050204ALUMCSM4220-3 & MED4220. The assembly procedure is described as follows.

Solder 28 AWG tinned Cu bare wire from Alpha Wire Company onto the contact pads with water-soluble solder from Kester.

Clean EFITT050203ALUMCSM4220-3 & MED4220 with



Warm tap H₂O, DI H₂O rinse, N₂ blow dry.

US in CTA1 (fresh solution) for 5min @ 50⁰C, DI H₂O rinse.

US in CTA2 (fresh solution) for 5min @ 50⁰C, DI H₂O rinse.

US in Vertrel MS992 Solvent & Flux Remover for 5min @ 50⁰C.

US in IPA for 5min @ 50⁰C.

Blow-dry with N₂, air dry under hood about 1 hr before VTES coating.

Clean EFITT050204ALUMCSM4220-3 & MED4220 with

US in CTA1 (used solution) for 5min @ 50⁰C, fresh CTA rinse.

US in CTA2 (used solution) for 5min @ 50⁰C, fresh CTA rinse.

US in CTA3 (used solution) for 5min @ 50⁰C, DI H₂O rinse.

US in CTA4 (fresh solution) for 5min @ 50⁰C, DI H₂O rinse

US in Vertrel MS992 Solvent & Flux Remover for 5min @ 50⁰C.

US in IPA for 5min @ 50⁰C.

Blow-dry with N₂, air dry under hood about 10 min before VTES coating.

Vinyltriethoxysilane(VTES) Adhesion Promoter Preparation

Mix 95ml ethanol with 5 ml DI H₂O.

Adjust PH to 4.5-5.5 by adding Acetic acid.

Stir in 2ml Vinyltriethoxysilane, sit for 5 min to allow hydrolysis and silanol formation.

Place sample into VTES solution and agitate for 2 min, then dip into ethanol briefly to remove excess VTES solution. Cure the adhesion promoter at 110⁰C for 5-10 min.



Slide Silastic tubing from Dow Corning (0.058"ID, 0.077"OD) over bare wires.

Coat EFITT samples with either CSM4220-3 (Lot# 34302) or old MED4-4220, TT first, leads & contacts next, and Silastic tube filling last. Old MED4-4220 acts like quick cure silicone. Lots of air bubbles trapped in the silicone during encapsulating process.

Cure silicone at 150⁰C for 3 hrs.

There are two differences in preparation between EFITT050203 and EFITT050204 devices. While EFITT050203 was coated with fresh prepared VTES solution and then sat under hood for overnight before encapsulated with silicone, EFITT050204 was coated with 1day old adhesion promoter and immediately encapsulated with silicone thereafter.

On 2/8/05, EFITT050203ALUMCSM4220-3&MED4220 and EFITT050204ALUMCSM4220-3&MED4220 were put under dry test. Two-day measurement showed all of the devices had resistance at 10^{11} to 10^{12} Ω . After PB ringer was added to the test tube on 2/11/05, EFITT050203ALUMCSM4220-3 only passed one cycle of leakage measurement at 10^{10} Ω with full sweep range (-5, +5V) before failing and EFITT050203ALUMMED4220 over ranged on the very first cycle. Measurement was terminated on 3/2/05. Microscopic study showed all the solder joints as well as TT was corroded on EFITT050203ALUMCSM4220-3, indicating general failure of CSM4220-3 protection. However, for EFITT050203ALUMMED4220, TT section was well protected, although half of solder joints were severely corroded. Unsealed air bubbles trapped during quick cure process of old MED4-4220 apparently caused the leakage. The pass test measurement for EFITT050204ALUMCSM4220-3 in PB Ringer lasted 3 days and EFITT050204ALUMMED4220 held 9 days before quickly dropped from $10^{10}/10^{11}$ Ω (-5, +5V) to over range. Microscopic examine showed no corrosion on the solder joints for CSM4220-3 device. However dendrite corrosion, apparently still caused by contamination of solder flux, was observed between inner two leads and the corrosion had extended onto TT. As for the device protected by MED4-4220, corrosion was found originated from a point on bare



wire and extended onto the solder joint. A further examine revealed that the failure was apparently coming from a small cut as well as trapped air bubbles in the silicone near the point of the bare 28AWG wire. In conclusion, comparing to CSM4220-3, MED4-4220 seems offer better protection to EFITT devices if air bubbles could be avoided. While vinyltriethoxysilane may help the adhesion of MED4-4220 onto the substrates, the effect of VTES is not clear for CSM4220-3 silicone. Pass test results are summarized in Table7 and further evaluation on silicone adhesion promoter will be continued.

EFITT050307ALUMED4-4220A, B, C

In this experiment, wires were soldered onto EFITT contact pads with water-soluble solder paste in Reflow Oven instead of by solder iron. The high temperature of solder iron may change the chemical structure of water-soluble solder and make the removal of solder flux more difficult. New MED4-4220 (Lot# 28806) was used for encapsulating the devices and vinyltriethoxysilane (VTES) coupling agent from Gelest Inc (Lot# 38-2520) was again applied for improving silicone adhesion on EFITT substrates. Three devices, EFITT050307ALUMED4-4220A&B&C, were assembled and assembly procedure are described as follows.

Reflow once 28AWG tinned Cu bare wires onto the contact pads of EFITT050307ALUMED4-4220A with water-soluble solder paste.

Reflow twice 28AWG tinned Cu bare wires onto the contact pads of EFITT050307ALUMED4-4220B with water-soluble solder paste.

Reflow twice pre-cleaned silicone insulated Bay wires onto the contact pads of EFITT050307ALUMED4-4220A with water-soluble solder paste.

Clean solder flux with

Warm Tap H₂O

US in CTA1 (used solution) for 1min @ 45⁰C, fresh CTA rinse.

US in CTA2 (used solution) for 1min @ 45⁰C, fresh CTA rinse.

US in CTA3 (used solution) for 1min @ 45⁰C, fresh CTA rinse.



US in CTA4 (fresh solution) for 1min @ 45⁰C, DI H₂O rinse.

US in DIH₂O for 1min @ 45⁰C, DI H₂O rinse.

US in Vertrel MS992 Solvent & Flux Remover for 1min @ 45⁰C.

US in IPA for 1min @ 45⁰C.

Blow-dry with N₂, air dry under hood about 10 min before VTES coating.

Vinyltriethoxysilane(VTES) Adhesion Promoter Preparation

Mix 95ml ethanol with 5 ml DI H₂O.

Adjust PH to 4.5-5.5 by adding Acetic acid.

Stir in 2ml Vinyltriethoxysilane, sit for 5 min to allow hydrolysis and silanol formation.

Place sample into VTES solution and agitate for 2 min, then dip into ethanol briefly to remove excess VTES solution. Cure the adhesion promoter at 110⁰C for 5-10 min. EFITT050307ALUMED4-4220A was coated with fresh VTES and EFITT050307ALUMED4220B&C with one-day-old VTES solution.

Slide Silastic tubing from Dow Corning (0.062"ID, 0.095"OD) over bare wires.

Fill the Silastic tube first and then coat EFITT samples with new MED4-4220, TT first, leads & contacts next.

Cure silicone at 150⁰C for 3 hrs.

Fill some of trapped air bubbles with MED4-4220, over coat the samples with MED4-4220; cure the silicone at 150⁰C for 3 hrs.

On 3/11/05, EFITT050307ALUMED4-4220A, B, C were put under dry test. Two-day measurement showed the devices A & B had resistance at 10¹¹ while device C at 10¹² Ω. After PB ringer was added to the test tube on 3/14/05, pass test for one-time reflowed bare-wire sample EFITT050307ALUMED4-4220A lasted three days before dropping from 9.7x10¹⁰ Ω (-5, +5V) to 4.9x10¹⁰ Ω (-2, +5V). Device was terminated on 3/22/05. Microscopic study after test revealed that silicone seemed not bond well near the contact region and corrosion products moved



freely between inner two solder joints. Corrosion product and salt crystals were also observed near the leads. A peel test further confirmed the poor adhesion between new MED4-4220 and EFITTALUM substrate since the silicone can be easily peeled off without any silicone residue left behind. Furthermore, it is found that new MED4-4220 still acts like quick cure silicone. Lots of air bubbles trapped in the silicone especially near the contact region during encapsulating process. Pass test for twice reflowed bare-wire sample EFITT050307ALUMED4-4220B lasted two days before suddenly dropping from $10^9 \Omega$ (-5, +5V) to over range, and twice reflowed sample with silicone insulated Bay wire EFITT050307ALUMED4-4220C failed on the first measurement at $4.9 \times 10^9 \Omega$ (0, +5V). After first week of measurement, devices B & C were taken out of solution and examined under microscope. TT section was well protected for both devices. Although the corrosion was observed on some solder joints for device B, no apparent corrosion was found on device C. Pass test was continued for another three weeks in order to show how the corrosion would progress. After terminating the test on 4/11/05, corrosion on TT as well as solder joints were observed on both devices B & C. Again, silicone was easily peeled off from the substrates without leaving residue behind, indicating poor adhesion of MED4-4220 on reflow-processed EFITTALUM substrates. Pass test results are summarized in Table 8.

EFITT050324ALUMCSM4220-3

The objective of this experiment is to reexamine rosin solder after length of trouble with effectively removing water-soluble solder flux. One triple tracks on alumina substrate, EFITT050324ALUMCSM4220-3, was assembled. The assembly procedure is described as follows.

28AWG tinned Cu bare wires were soldered onto EFITT contact pads with rosin solder at low temperature of 575°C .

Rosin solder flux was cleaned by

Spray with MS-795 Heavy Duty Solvent & Flux Remover.

US in Vertrel XMS Plus1 (MS-797) for 1 min. Spray with MS-755.



US in Vertrel XMS Plus 2 (MS-797) for 1 min. Spray with MS-755.

US in DIH₂O for 1min. DI H₂O rinse.

US in IPA for 1min. Blow-dry with N₂. Bake at 150⁰C for 5 min.

Slide Silastic tubing from Dow Corning (0.058"ID, 0.077"OD) over bare wires.

Coat EFITT sample with CSM4220-3 (Lot# 34302), TT first, leads & contacts next, and Silastic tube filling last.

Cure silicone at 150⁰C for 3 hrs.

On 3/28/05, EFITT050324ALUMCSM4220-3 was put under dry test. Two-day measurement showed the device had resistance at 10¹¹ Ω. On 3/30/05, PB ringer was added to the test tube, and pass test for this rosin-solder processed EFITTALUM device lasted only one cycle at 1.5x10⁹ Ω (-5, +5V) before over range. After terminating the test on 4/11/05, EFITT050324ALUMCSM4220-3 was examined under microscope. It is observed that part of TT as well as leads were corroded, yet no corrosion on any of the solder joints. Again, silicone was found easily peeled off from the substrates without leaving residue behind, indicating poor bond between CSM4220-3 and rosin-solder processed EFITTALUM substrate. Pass test result is summarized in Table 9.

In the period of January through March 2005, three sets of inter digital electrodes (SmallLCPIDEHFS040720, LongLCPIDEHFS040720, QuartzIDEHFS040720) coated with hot filament deposited silicone from MIT were also examined. Each device was glued on a microscope slice which was then slid through a precut slot on a test jar's green cap and was fixed with quick cure silicone at a position such that contacts were outside of test jars to avoid the problem of silicone adhesion at contact region due to solder flux contamination. The experiment procedures and results up to March 2005 are summarized as follows.



Testing of CVD Hot Filament Silicone depositions

QuartzIDEHFS040720

Four IDE on quartz were assembled into a test jar. Dry test showed the devices E4 & E10 had resistance of 10^{10} and $10^{11}\Omega$ respectively, and E6 & E8 at $10^{13}\Omega$. After the test jar was filled with Phosphate Buffered Ringer (PBR) on 1/12/05, the resistance of E4 has gradually increased from 6.6×10^9 to $1.7 \times 10^{11}\Omega$, devices E10 continues at $10^{10}\Omega$ level, and E8 remains at $10^{12}\Omega$. Yet E6 failed on the first wet test, apparently due to pinhole defect in the coating. The resist data are summarized in Table3 and pass test is continuing.

On 1/7/05, the measurement for 25 μ m D Au wires loops coated with dense PTFE NP by GVD from Dr. Gleason were terminated. The samples were examined under microscope. The experiment procedures and results are summarized as follows.

Teflon coated Au wire was assembled into four wire loops through pairs of holes on the caps of the testing jars. The Teflon coating on two ends of the wire loop was burned off with a micro-torch for contacts. The ends of Au wire were then wrapped around 30AWG Belden wires that led to connector for pass test measurement. The loose connected joints of Au and Belden wires were further secured with H20E Ag epoxy from Epoxy Technology. The assemblies were finally ready to test.

On 11/19/04, MITAUGVDNPB101504AFE04, E06, E08, E10 were under dry test. Two-day measurement showed that all the devices had resistance at 10^{13} to $10^{14}\Omega$. However, after Phosphate Buffered Ringer solution was added to the test jars on 11/22/04, three out of four devices (E04, E06, E08) immediately showed high leakage current with a resistance as low as $10^9/10^8\Omega$ and extremely limited sweeping range of (0, +1V). Device E10 lasted one month wet test and was measured at $7 \times 10^{12}\Omega$ with full sweeping range of (-5, +5V) before it also failed.



The electrical measurement for MITAUGVDNPB101504AF is summarized in Table1.

On 1/7/05, devices were taken out PB ringer and examined under microscope. It is found that all the wire loops were covered with white salt crystals, Apparently PTFE coatings on Au were porous enough to support electroplating of salts onto the wires. Moreover, all the wire loops were not broken when they were taken out of the Ringer solution; however, when E4 was rinsed with DI H₂O, the loop broke. Pictures showed sharpened tip at the broken ends indicating pinhole failure in the PTFE coating.

In March 2005, half of the RSHFV3D3TBP samples were terminated due to poor pass test performance. The failed samples were thoroughly examined under microscope thereafter. The experiment procedures and results up to March 2005 are summarized in follows.

Four sets of hot filament deposited silicone film on Si substrates from V₃D₃+TBP at two different conditions were initially examined under microscope. After assembly, the samples were put under dry test for few days and then were added saline solution to begin wet test at 37⁰C on 6/1/04. Pictures of surface morphologies are taken and saved at \aa cleanroom\LabPics\RingSquare\RSHFV3D3TBP. The pass test results up to March 2005 are summarized in Table 1.

RSHFV3D3TBP041204A(E4, E6, E8, E10)

1μ film deposited at condition#1(P=300mT, T_f=500⁰C, T_s=60⁰C) has the surface morphology of “star field”. The resistance measurement shows that in general RSHFV3D3TBP041204A has poor hermetic property. After soaking, resistance of all four devices quickly dropped from 10⁹Ω to 10⁸Ω and the measurements were swept at limited range. The pass test was terminated on 3/10/05.

Microscopic study after pass test showed extensive corrosion throughout the test surfaces, indicating general failure of the coating.



RSHFV3D3TBP041204B1(E4, E6, E8, E10) & RSHFV3D3TBP041204B2(E4, E6, E8, E10)

5 μ film deposited at condition#1 ($P=300\text{mT}$, $T_f=500^\circ\text{C}$, $T_s=60^\circ\text{C}$) has the “wrinkled” surface morphology. As indicated in Table1, after nine months soaking, five out of eight devices keep the resistance at $10^{12}\Omega$ while the rest at $10^9\Omega$. All the measurements for 5 μ film were swept at full range (-5, +5V) and the pass test is continuing.

RSHFV3D3TBP041404(E4, E6, E8, E10)

1 μ film deposited at condition#2 ($P=300\text{mT}$, $T_f=400^\circ\text{C}$, $T_s=60^\circ\text{C}$) has a very smooth surface. Unfortunately, due to the trouble in test system, the data for the first three and half month (6/1/04-10/19/04) cannot be trusted. Since 11/17/04, the resistances of all four devices have continued mostly at $10^9\Omega$. While the measurements for E4 & E6 keep at full sweep (-5, +5V), the range for devices E8 & E10 has deteriorated to (-5, +3V). The pass test was terminated on 3/16/05. Microscopic analysis after pass test revealed interesting corrosion pattern that is either like a contour map or similar to pattern of clouds. A further magnified picture showed the contours are made of series of small-bead-like corrosion sites. The pictures were taken and saved in the file of RSHFV3D3TBP041404.

In March 2005, several pass tests on RSHFS040720 samples were terminated due to high leakage measurement. The failed samples were thoroughly examined under microscope thereafter, and the experiment procedures and results up to March 2005 are summarized in follows.

Two sets of ring square sample with hot filament deposited silicone film on Si substrates from MIT were assembled, examined under microscope, and then directly put in PB Ringer for wet test at 90°C on 11/29/04. Microscopic examine showed the samples have the surface morphology of flower shaped clusters about 30 μ in size throughout the film. Pictures are taken and saved at \aa



cleanroom\LabPics\RingSquare\RSHFS040720. The pass test results are summarized in Table 1.

RSHFS040720A(E4, E6, E8, E10)

After nearly three and half month measurement, the pass test shows that resistance of E6 & E10 remains at $10^{10}\Omega$ and $10^9\Omega$ respectively with full sweep range of (-5, +5V). On the other hand, resistance of E4 & E8 quickly dropped from $10^{10}\Omega$ (-5, +4V) to $10^9\Omega$ after the first measurement with a limited sweep range of (-5, +1V). The pass test for E4 & E8 was terminated on 3/11/05.

Microscopic examine after the pass test showed one site of plane (1,1,1) corrosion on each sample, apparently caused by pinhole defect in the films. The study also revealed the massive cracking in the film. Since there is no evidence of corrosion occurred at cracks, the film apparently was cracked after E4&E8 were taken out of ringer solution, rinsed with DIH₂O, and air dried over weekend before the examine was taken place. Pass test measurements for E6 & E10 are continuing.

RSHFS040720B(E4, E6, E8, E10)

As indicated in Table1, although device E4 started at $10^9\Omega$ (-5, +5V), its sweep range suddenly dropped to (-5, +2V) after 19 days measurement, indicating that the process of leakage was taken place. The resistance of E10 also started at good level of $10^{10}\Omega$ with a full sweep range of (-5, +5V) and then quickly dropped to $10^9\Omega$ within the first week of measurement at a limited sweep range of (-5, +1V). The pass test for both devices was terminated on 3/10/05. Microscopic study after the test showed two sites of plane (1,1,1) corrosion on E4 and multiple sites for E10. After two month and three week measurement, the resistance of E6 suddenly dropped from $10^{10}/10^9\Omega$ (-5, +5V) to $2.7 \times 10^8\Omega$ (-2, +1V). E6 was also terminated on 3/10/05. The failure for E6 however was not caused by plane (1,1,1) corrosion due to pinhole defects in the film; instead, salt crystal was observed under the ring at 7-9 o'clock as well as backside of Si substrate at the associate location. A further microscopic study revealed that the broken air bubbles trapped in the silicone used for glue the ring onto the



substrate caused the leakage to the Si substrate. The last device to be discussed in RSHFS040720B is E8. Pass test shows that resistance of E8 began at $10^{10}\Omega$ and has gradually increased to $10^{11}\Omega$ after nearly three and half month testing with a full sweep range of (-5, +5V). The pass test measurement for E8 is currently continuing.



MITSilicone CVD Depositions

January – March 2005

Work over the past months has focused on three areas:

Deposition and characterization of new homopolymers for future copolymer use.

Preparation of samples for film mechanical property testing.

Presentation of polymer chemical synthesis at the American Chemical Society national meeting.

In order to improve the depositional characteristics of the novel polymer films under development, we are searching for a secondary siloxane precursor with which to copolymerize. The goal of the copolymerization would be to retain the excellent physical properties of the current films while reducing the steric hindrance of the polymerization reaction to allow for faster deposition at lower filament temperatures. To this end, three comonomers have been investigated (see attached table). To test the viability of these monomers, as well as collecting data for comparison to any future copolymer, homopolymers of each of these three species have been deposited and analyzed using ellipsometry and FTIR. The best candidate appears to be 1,5-

DIVINYLHEXAMETHYLTRISILOXANE, based on depositional characteristics and required reactor conditions for polymerization. Both of the other monomers required reactor pressures much higher than those utilized for the current film depositions, and were therefore non-ideal candidates. Work in the near term will now move toward optimization of deposition conditions for copolymerization.


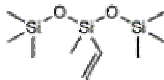

A novel methodology for testing the modulus of thin films has recently been described in the literature. The technique (NATURE MATERIALS 3 (8): 545-550 AUG 2004) utilizes a thin film of the material to be tested on top of a thicker block of PDMS. When the PDMS is stressed, the thin film will deform in a wave-like pattern due to the difference in physical properties between the two materials. The periodicity of the waves can then be used to calculate the ratio of the modulus of the film vs. the PDMS substrate. This technique is much easier to



perform than nano-indentation, but does require the deposition of a film onto a PDMS sample. Due to the process conditions required for deposition of our novel material, the films cannot be deposited directly on the PDMS substrate as the process would likely modify the PDMS physical properties. Instead, films must be deposited on another substrate and then transferred to the PDMS. Over the last few months, samples have been prepared through deposition of thin films on Teflon coated substrates and then transfer to PDMS. This process, however, has had some difficulties as the films will often not transfer fully. Work on these measurements will continue, with the possible use of a sacrificial layer between the substrate and the thin film to allow for easier transfer of the material.

In addition to the work outlined above, results from the research were presented at the American Chemical Society national meeting in March. The presentation centered on the chemical reaction mechanism of the polymerization and the excellent control over the chemical pathways.



Name	Structure	CAS #	Vapor Pressure
1,5-DIVINYLHEXAMETHYLTRISILOXANE		136777-27-0	20 Torr @ 80C
VINYLMETHYLBIS(TRIMETHYLSILOXY)SILANE		5356-85-4	760 Torr @ 167C
VINYLPENTAMETHYLDISILOXANE		1438-79-5	760 Torr @ 120C